Version 1.2, Nov 2002

**OLIN-WILMINGTON**
**LEVEL I DATA QUALITY EVALUATION**
**STANDARD OPERATING PROCEDURE AND CHECKLIST**
**ICP METALS BY METHOD 6010B/200.7**
Al, Cr, Fe only

Reviewer/Date Chris Ricardi 3/17/10
Sr. Review/Date
Lab Report # 25553
Project # 6107100016

## 1.0 Laboratory Deliverable Requirements

No Qual

**1.1 Laboratory Information**: Was all of the following provided in the laboratory report? Yes [✓] No [ ] N/A [ ] Comments:
Check items received.

☑ *Name of Laboratory* ☑ *Address* ☑ *Project ID* ☑ *Phone #* ☑ *Sample identification – Field and Laboratory*
Client Information: ☑ *Name* ☑ *Address* ☑ *Client Contact* *(IDs must be cross-referenced)*

**ACTION**: If no, contact lab for submission of missing or illegible information.

***1.2 Laboratory Report Certification Statement*** ***Yes [✓]*** *No [ ] N/A [ ] Comments:*

*Does the laboratory report include a completed Analytical Report Certification in the required format?*

***ACTION**: If no, contact lab for submission of missing certification or certification with correct format.*

***1.3 Laboratory Case Narrative:*** ***Yes [✓]*** *No [ ] N/A [ ] Comments:*

☐ *Narrative serves as an exception report for the project and method QA/QC performance.* ☑ *Narrative includes an explanation of each discrepancy on the*

*Certification Statement.*

***ACTION**: If no, contact lab for submission of missing or illegible information.*

**1.4 Chain of Custody (COC)** copy present with all documentation completed Yes [✓] No [ ] N/A [ ] Comments:

**NOTE:** Olin receives and maintains the *original COC.*

**ACTION**: If no, contact lab for submission of copy of completed *COC.*

**1.5 Sample Receipt Information (*Cooler Receipt Form present?*):** Yes [✓] No [ ] N/A [ ] Comments:

Were each of the following tasks completed and recorded upon receipt of the sample(s) into the laboratory?

☑ Sample temperature confirmed: must be 1° – 10° C. (If samples were sent by courier and delivered on the same day as collection, temperature requirement does not apply).

☑ Container type noted ☑ sample condition observed ☐ pH verified (where applicable) ☑ Field and lab IDs cross referenced

**ACTION**: If no, contact lab for submission of missing or incomplete documentation.

**1.5.1** Were all samples delivered to the laboratory without breakage? **Yes** [✓] No [ ] N/A [ ] Comments:

**1.5.2** Does the *Cooler Receipt Form* or Lab Narrative indicate other problems with sample receipt, condition of the samples, analytical problems or special circumstances affecting the quality of the data? Yes [ ] **No** [✓] N/A [ ] Comments:

**1.6 Sample Results Section:** *Was each of the following requirements supplied in the laboratory report for each sample?* **Yes** [ ] No [ ] N/A [ ] Comments:

☑ Field ID and Lab ID ☑ *Date and time collected* ☑ Analyst Initials ☑ *Dilution Factor* ☑ % moisture or solids ☑ *Reporting limits*
N/A ☐ *Clean-up method* ☑ Analysis method ☑ *Preparation method* ☑ *Date of preparation/extraction/digestion clean-up and analysis, where applicable*
☑ Matrix ☑ Target analytes and concentrations ☑ Units (soils must be reported in dry weight)

**ACTION**: If no, contact lab for submission of missing or incomplete information.

**1.7 QA/QC Information:** Was each of the following information supplied in the laboratory report for each sample batch? **Yes** [✓] No [ ] N/A [ ] Comments:

**OLIN CORPORATION**
**LEVEL I DATA QUALITY EVALUATION – OPTION 1**
**STANDARD OPERATING PROCEDURE AND CHECKLIST**
**ICP METALS BY METHOD 6010B/200.7**

☑ Method blank results ☑ LCS recoveries ☑ MS/MSD recoveries and RPDs ☐ Laboratory duplicate results (where applicable)

**ACTION**: If no, contact lab for submission of missing or incomplete information. See LCSD, MSD

## 2.0 Holding Times

Have any technical holding times, determined from date of collection to date of analysis, been exceeded? Holding time for metals is 180 days from sample collection to analysis for both water and soil. Yes [ ] No [✓] N/A [ ] Comments:

**NOTE:** List samples that exceed hold time with # of days exceeded on checklist

**ACTION:** If technical holding times are exceeded, qualify all positive results (J) and non-detects (UJ). If grossly exceeded (2X holding time) reject (R) all non-detect results.

## 3.0 Laboratory Method

**3.1** Was the correct laboratory method used? **Yes [✓]** No [ ] N/A [ ] Comments:

| | |
|---|---|
| Water Digestion | 3005A or 3010A or 3020A |
| Soil Digestion | 3050B |
| Metals | 6010B or 200.7 |

**ACTION**: If no, contact laboratory to provide justification for method change compared to the requested method. Contact senior chemist to inform Client of change and to request variance.

**3.2** Are the practical quantitation limits the same as those specified by the ☐ SOW ☑ QAPP ☐ Lab ☐ MADEP **Yes [✓]** No [ ] N/A [ ] Comments:

**NOTE:** *Verify that the reported metals match the target list specified on the COC.*

**ACTION**: If no, evaluate variation with respect to sample matrix, preparation, dilution, moisture, etc. If sample PQL is indeterminate, contact lab for explanation.

3.3 Are results present for each sample in the SDG? Yes [✓] No [ ] N/A [ ] Comments:

**ACTION:** If no, check Request for Analysis to verify if method was ordered and COC to verify that it was sent, and contact lab for resubmission of the missing data

3.4 If dilutions were required, were dilution factors reported? Yes [✓] No [ ] N/A [ ] Comments:

**ACTION:** If no, contact the lab for submission.

## 4.0 Method Blanks

4.1 Is the Method Blank Summary present? Yes [✓] No [ ] N/A [ ] Comments:

**ACTION**: If no, call the laboratory for submission of missing data.

4.2 Frequency of Analysis: Was a method blank analyzed for each digestion batch of < 20 field samples? Yes [✓] No [ ] N/A [ ] Comments:

**ACTION**: If no, contact laboratory for justification. Consult senior chemist for action needed. Narrate non-compliance.

4.3 Is the method blank less than the PQLs for all target elements? Yes [ ] No [✓] N/A [ ] Comments: Fe 1.56 mg/kg

**NOTE**: *MADEP requires the method blank to be matrix matched and digested with the samples*

4.4 Do any method blanks have positive results for metals? Qualify data according to the following: Yes [✓] No [ ] N/A [ ] Comments: All values in Samples much higher. No Qual

(*) Lab B quals removed

If the sample concentration is < 5 × blank value, flag sample result non-detect "U" at the PQL or the concentration reported if greater than the PQL.

If the sample concentration is > 5 × blank value, no qualification is needed.

**ACTION:** For any blank with positive results, list all contaminants for each method blank including the concentration detected and the flagging level (flagging level = 5x the blank value) and the associated samples and qualifiers.

## 5.0 Laboratory Control Standard

**5.1** Was a laboratory control standard run with each analytical batch of 20 samples or less? **Yes** [✓] No [ ] N/A [ ] Comments:

**NOTE:** *A full target, second source LCS is required by MADEP.*
**ACTION**: Call laboratory for LCS form submittal. If data are not available, use professional judgement to evaluate data accuracy associated with that batch.

**5.2** Is a LCS Summary Form present? **Yes** [✓] No [ ] N/A [ ] Comments:

**ACTION**: If no, contact lab for resubmission of missing data.

**5.3** Is the recovery of any analyte outside of MADEP control limits? Yes [ ] **No** [✓] N/A [ ] Comments:

| Sample Type | ***MADEP % Rec*** |
|---|---|
| Water | *80-120* |
| Soil | within Lab generated limits |

**ACTION:** If recovery is above the upper limit, qualify all positive sample results within the batch as (J). If recovery is below the lower limit, qualify all positive and non-detects results within the batch as (J). If LCS recovery is <30%, positive and non-detect results are rejected (R).

Comments:

## 6.0 Matrix Spikes

Matrix spikes may be collected at different frequencies based on monthly, quarterly, or task specific schedules. Confirm spike requirements for each set with the senior chemist.

**6.1** Were project-specific MS/MSDs collected? List project samples that were spiked.

Yes [✓] No [ ] N/A [ ] Comments:

**ACTION**: If no, contact senior chemist to see if any were specified.

**6.2** Is the Matrix Spike/Matrix Spike Duplicate Recovery Form present?

Yes [✓] No [ ] N/A [ ] Comments:

**NOTE:** *A full target, second source MS/MSD is required by MADEP.*
**ACTION:** If any matrix spike data are missing, call lab for resubmission.

**6.3** Were matrix spikes analyzed as indicated on the COC and project schedule?

Yes [✓] No [ ] N/A [ ] Comments:

**ACTION**: If any matrix spike data are missing, call lab for resubmission. If none, no qualification is needed. Narrate non-compliance.

**6.4** Are any metal spike recoveries outside of the QC limits?

Yes [✓] **No** [ ] N/A [ ] Comments:

Al, Fe > 4X spike. No evaluation Possible.

| Sample Type | ***MADEP*** ***% Rec*** | QAPP % Rec | Method |
|---|---|---|---|
| Water | *75-125* | N/A | 6010B |
| Water | *N/A* | 70-130 | 200.7 |
| Soil | *75-125* | 75-125 | 6010B |

**NOTE**: $\%R = \frac{(SSR-SR)}{SA} \times 100\%$ Where: SSR = Spiked sample result, SR = Sample result, SA = Spike added

Cr - ok

**NOTE:** If dilutions are required due to high sample concentrations (> 4X spike), the data are evaluated, but no flags are applied.

**NOTE:** If only one of the recoveries for an MS/MSD pair is outside of the control limits, no qualification is necessary. Use professional judgment for the MS/MSD flags.

**ACTION:** MS/MSD flags only apply to the sample spiked. If the recoveries of the MS and MSD exceed the upper control limit, qualify positive results as estimated (J). If the recoveries of the MS and MSD are lower than the lower control limit, qualify positive results and non-detects (J).

**6.5** Are any RPDs for MS/MSD recoveries outside of the QC limits? Yes [ ] **No** [✓] N/A [ ] Comments:

**NOTE:** $RPD = \frac{S-D}{(S+D)/2} \times 100\%$ Where: S = MS sample result
D = MSD sample result

**NOTE:** If dilutions are required due to high sample concentrations, the data are evaluated, but no flags are applied.

**ACTION:** If the RPD exceeds the control limit, qualify positive results and non-detects (J).

## 7.0 Laboratory Duplicate

**7.1** Was a laboratory duplicate sample analyzed? If so, is the Laboratory Duplicate Sample Form present? **Yes** [ ] No [ ] N/A [✓] Comments:

***NOTE:*** *MADEP refers to this sample as a "matrix duplicate".*

**ACTION:** If not analyzed, qualification is not needed. If data is missing, contact laboratory for resubmission of report. Narrate non-compliance.

**7.2** Is the RPD between the result for the laboratory duplicate sample and the result for the parent sample outside of the QA/QC limits? Yes [ ] **No** [ ] N/A [✓] Comments:

| *MADEP Laboratory Duplicate Sample RPD Criteria:* | QAPP RPD |
|---|---|
| *For aqueous results > 5× RL, RPD must be ± 20%* | 20 |
| *For aqueous results < 5× RL, RPD must be ≤ RL* | 20 |
| *For soil/sediment results > 5× RL, RPD must be ± 35%* | 20 |
| *For soil/sediment results < 5× RL, RPD must be ≤ 2× RL* | 20 |

**ACTION**: If the RPD exceeds the limits, qualify both positive results and non-detects as estimated and flag them J. Narrate non-compliance

## 8.0 Sampling Accuracy

The majority of ground water samples are collected directly from a tap, process stream, or with dedicated tubing. Rinse blanks will not be collected.

8.1 Were rinsate blanks collected? Prior to evaluating rinsate blanks, obtain a list of the associated samples from the senior chemist. Yes [ ] No [✓] N/A [ ] Comments:

8.2 Do any rinsate blanks have positive results? Yes [ ] No [ ] N/A [✓] Comments:

***NOTE**: MADEP does not require the collection of rinsate blanks.*

**ACTION:** Evaluate rinsate results against blank results to determine if contaminant may be laboratory-derived. If results are not lab-related, qualify according to below.

If the sample concentration is < 5 × blank value, flag sample result non-detect "U" at the PQL or the concentration reported if greater than the PQL.

If the sample concentration is > 5 × blank value, no qualification is needed.

## 9.0 Field Duplicates

**9.1** Were field duplicate samples collected? Obtain a list of samples and their associated field duplicates. Yes [✓] No [ ] N/A [ ] Comments:

9.2 Were field duplicates collected per the required frequency? Yes [✓] No [ ] N/A [ ] Comments:

SOW ☐ QAPP *(1 per 10)* ☑ *MADEP Option 1 (1 per 20)* ☐ *MADEP Option 3 (1 per 10)* ☐

**9.3** Was the RPD ≤ 50% for soils or waters? Calculate the RPD for all results and attach to this review. **Yes** [✓] No [ ] N/A [ ] Comments: See attached

**ACTION**: RPD must be ≤50% for soil and water. Qualify data (J) for both sample results if the RPD exceeds 50%.

**10.0** **Special QA/QC**

**10.1** Were both total and dissolved metals analysis performed? If so, the dissolved metal concentration should not exceed that of the total metal. Yes [ ] **No** [ ] N/A [✓] Comments:

**ACTION:** If results for both total and dissolved are ≥ 5x the PQL **and** the dissolved concentration is 10% higher than the total, flag both results as estimated (J). If total and dissolved concentrations are less than 5x the PQL **and** the **difference** exceeds 2x the PQL, flag both results as estimated (J)

**10.0 Application of Validation Qualifiers**

Was any of the data qualified? Yes [ ] No [✓] N/A [ ] Comments:

If so, apply data qualifiers directly to the DQE copy of laboratory report and **flag pages** for entry in database.

## REFERENCES

LAW, 1999, "Final Quality Assurance Project Plan, Olin Wilmington Property, 51 Eames Street, Wilmington, MA", LAW Engineering and Environmental Services, Kennesaw, GA 30144. August 1999

U.S. Environmental Protection Agency (USEPA), 1989. "Region 1 Laboratory Data Validation Functional Guidelines For Evaluating Inorganic Analyses"; Hazardous Site Evaluation Division; February 1989.

MADEP, 2001. Massachusetts Department of Environmental Protection Bureau of Waste Site Cleanup, "Massachusetts Quality Assurance and Quality Control (QA/QC) Requirements." BWSC-CAM, Interim Final Draft, Revision No. 2, 5 October 2001.

MADEP, 2001. Massachusetts Department of Environmental Protection Bureau of Waste Site Cleanup, "Quality Assurance and Quality Control Guidelines for Sampling, Data Evaluation and Reporting Activities," BWSC-CAM, Section VII, Public Comment Draft, Revision No. 0, 21 December 2001.

| | | Sample | | Dup | | RPD |
|---|---|---|---|---|---|---|
| OC-SD-SD-SD1-0.0/0.5 | Percent Moisture | 44 | | 42 | | 5 |
| OC-SD-SD-SD1-0.0/0.5 | Percent Solids | 56 | | 58 | | -4 |
| OC-SD-SD-SD1-0.0/0.5 | Aluminum | 9200 | | 11000 | | -18 |
| OC-SD-SD-SD1-0.0/0.5 | Chromium | 21 | | 22 | | -5 |
| OC-SD-SD-SD1-0.0/0.5 | Iron | 11000 | B | 18000 | B | -48 |